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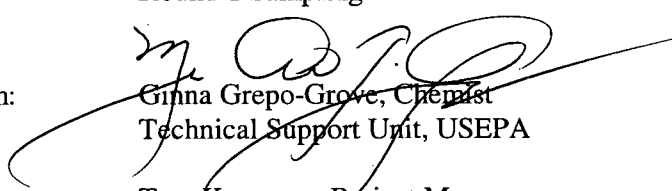
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

September 30, 2003

Reply to
Attn of: OEA-095

MEMORANDUM

Subject: Data Validation Report for Semi-volatile Organic Compounds (SVOCs) full scan with Tentatively identified Compound (TICs), SVOCs using Selected Ion Monitoring (SIM) for low detection limit Polynuclear Aromatic Hydrocarbons (PAHs) and other SVOCs, Pesticide (Pest), PCB Aroclors (PCBs), Butyl Tins, Metals, and Percent Lipids (% lipids) Analyses of Fish and Clam Tissue Samples Collected for the Portland Harbor RI/FS Round 1 Sampling Event

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The quality assurance (QA) review of the analytical data generated from the analysis of 23 fish and 2 clam tissue samples collected from the above referenced site has been completed. The SVOC full scan and SVOC SIM analyses were performed by Analytical Resources, Inc (ARI) located in Tukwila, WA. The rest of the analyses, i.e., pests, PCBs, butyl tins, metals, and % lipids were performed by Columbia Analytical Services (CAS) located in Kelso, WA. The two clam tissue samples were analyzed for butyl tins only. Table 1 lists the samples that were evaluated in this validation report and the collection date and verified time of sample receipt (VTSR) in the labs.

Table1 -Sample Numbers, and Dates of Sample Collection and VTSRs

Sample Number	ARI Lab Numbers	CAS Lab Numbers	Collection Date	VTSR at ARI	VTSR at CAS
LWG01FZ0609TSBBWBC10	-	K2300044-001	10/07/02	-	12/23/02
LWG01FZ0609TCBBWBC20	-	K2300044-002	10/03/02	-	12/23/02
LWG01FZ0609TSBBWBC30	-	K2300044-003	10/02/02	-	12/23/02
LWG01FZ0306TSBBWBC10	-	K2300044-04	09/24/02	-	12/23/02

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Sample Number	ARI Lab Numbers	CAS Lab Numbers	Collection Date	VTSR at ARI	VTSR at CAS
LWG01FZ0306TSBBWBC20	-	K2300044-005	09/24/02	-	12/23/02
LWG01FZ0306TSBBWBC30	-	K2300044-006	09/23/02	-	12/23/03
LWG0106R002TSSPWBC20	-	K2300044-007	10/07/02	-	12/03/02
LWG0106R004TSSPWBC00	-	K2300044-008	09/24/02	-	12/03/02
LWG0107R003TSSPWBC00	-	K2300044-009	08/16/02	-	12/03/02
LWG0107R006TSSPWBC00	-		11/07/02	-	12/03/02
LWG0108R002TSSPWBC00	-	K2300044-012	K2300044-010		12/03/02
LWG0108R003TSSPWBC00	-	K2300044-013	10/08/02		12/03/02
LWG0109R001TSSPWBC00	-	K2300044-014	08/13/02		12/03/02
LWG0103R001TSSPWBC00	FA37A	K2300044-015	07/23/02	12/02/02	12/03/02
LWG0103R002TSSPWBC10	FA37B	K2300044-016	09/03/02	12/02/02	12/03/02
LWG0105R001TSSPWBC00	FA37C	K2300044-017	7/31/02	12/02/02	12/03/02
LWG0109R002RSSPWBC00*	FA37D	K2300044-018	08/15/02	12/02/02	12/03/02
LWG0106R002TSSPWBC10	FA37E	K2300044-019	08/09/02	12/02/02	12/03/02
LWG01FZ0609TSBBWBC10MS	-	KWG0304486-1	10/07/02	-	12/23/02
LWG01FZ0609TSBBWBC10DM	-	KWG0304486-2	10/07/02	-	12/23/02
LWG0103R014TSLSWBC10	FA39A	-	07/22/02	12/02/02	-
LWG0108R010TSLSWBC00	FA39B	-	08/12/02	12/02/02	-
LWG0105R006TSLSWBC00	FA39C	-	07/29/02	12/02/02	-
LWG01FZ0609TSLSWBC00	FA39D	-	08/07/02	12/02/02	-
LWL0109R006TSLSWBC00	FA39E	-	08/14/02	12/02/02	-
LWG0106R002TSCAWBC00	-	K2303135-001	10/24/02	-	04/28/03
LWG0107R006TSCAWBC00	-	K2303135-003	11/12/02	-	04/28/03
LWG0106R002TSCAWBC00MS	-	KWG0306866-	10/24/02	-	04/28/03
LWG0106R002TSCAWBC00D	-	KWG0306866-2	10/24/02	-	04/28/03

* "RS" is not included in the sample matrix code defined in the FSP. Also this sample is not listed in the chain-of-custody documentation. This sample needs to be verified. The sample name could be LWG0109R002TSPPWBC00.

Table 2 lists the suite of parameters, clean-up and analytical methods used in the analyses:

Parameters	Methods	
	Clean-ups	Analysis
SVOC SIM (PAHs)	3640A; 3630A	SW846 - Modified Method 8270C SIM
SVOCs (full scan w/ TICs)	3640A	SW846 - Method 8270C
PCB (Aroclors)	3640A; 3620C; 3665A	SW846 - Method 8082
Pesticides	3640A; 3620C	Analysis: SW846- Method 8081A
Butyl Tins	Stallard	GC/FPD
Metals including mercury		SW 846 Methods: 6010, 6020, 7740 and 7471 A
% lipids		prep - Method 3540. PSEP analysis

DATA QUALIFICATIONS

The following comments refer to the laboratories' performance in meeting the Quality Control specifications outlined in the Quality Assurance Project Plan, the laboratory's Standard Operating Procedures, the analytical methods listed above and the Contract Laboratory Program's National Functional Guidelines for Organic and Inorganic Data Review. Some of the data quality elements were qualified using the reviewer's professional judgment.

The conclusions presented herein are based on the information provided for the review.

Chain-of Custody (COC) Records

The COC documentation for the samples listed above were complete except for sample LWG0109R002RSSPWBC00 (K2300044-018) reported by CAS. This sample is not listed in the COCs submitted with the data packages. This sample number needs to be verified and corrected because the FSP does not include an "RS" code for sample matrix.

Sample Homogenization and Condition upon Receipt

Axys Analytical Services (Axys) homogenized all of the tissue samples, distributed the aliquots and shipped them to ARI and CAS for subsequent analysis. The tissue samples were received in the laboratories frozen. The temperature blank in each cooler registered surface temperatures ranging from 2-4 °C. There were no problems encountered during sample receipt.

Holding Time - Acceptable

All of the samples were received frozen by the laboratory. Each of the sample container was stored

frozen at -20C in individual Ziploc bags until homogenization, extraction and analysis. All of the samples met both the extraction and analytical holding times criteria specified in the QAPP and the methods for each suite of parameters. None of the data were qualified on this basis.

Sample Preparation

All of the samples were prepared in accordance with the methods/SOPs specified in the QAPP. All of the samples for organic analyses went through the initial GPC clean-up. Additional clean-up techniques and multiple analyses were employed for each suite of organic parameters to remove interferences and achieve lower reporting limits. None of the data were qualified on this basis.

Target Compounds and Reporting Limits

All of the target compounds were reported on a wet-weight basis and were adjusted for sample amount extracted and dilution factors.

Metals: All of the metals target analytes were reported at a higher reporting limits (MRLs) than the MRLs specified in the QAPP. Only copper, manganese, nickel and zinc met the EPA established ACGs listed in the QAPP.

Pesticides: All of the pesticide MRLs met the MRLs listed in the QAPP with the exception of the following compounds reported at higher MRLs: beta-BHC, dieldrin, endosulfan II, 2,4'-DDD, cis-nonachlor and toxaphene. Also, some of the MRLs reported were raised by the reviewer due to interferences caused by other organic material and PCBs native to the samples

Larger sample amounts, special extract clean-ups and large volume injectors were used by the labs to get their reporting limits about 10x lower than the commonly used CLP reporting limits. Even with these sample processing modifications, only the compounds endosulfan I, endrin, hexachlorobutadiene, methoxychlor, mirex and hexachloroethane's MRLs met the tissue ACGs listed in the QAPP for pesticides.

Hexachlorobutadiene, hexachlorobenzene and hexachloroethane were also analyzed as part of the SVOC-SIM target list. It is recommended that data users utilize the hexachlorobenzene and hexachlorobutadiene results from the pesticide analysis.

PCBs as Aroclors: The PCBs were reported at the MRLs specified in QAPP. None of the QAPP-specified ACGs for Aroclors 1242, 1248, 1254 and 1260 were met.

SVOCs: All of the samples went through a series of fractionation clean-ups developed by the lab for this project. All of the compounds listed for SVOCs analysis in the QAPP were analyzed by the lab using full scan Method 8270C. The following compounds were added by the lab to the SVOC list: 2,3,4,6-tetrachlorophenol, 2,3,4,5-tetrachlorophenol, 2,3,5,6-tetrachlorophenol and 1,2-diphenylhydrazine (reported as Azobenzene) as these compounds could not be analyzed using 8270-SIM as specified in the QAPP because they are stripped and lost during the 8170-SIM silica gel clean-up process. Benzoic acid, aniline and isophorone were also stripped off during the clean-up processes and were not reported as target analytes by the lab.

PCBs: Five concentration levels of Aroclors 1016, 1260 and surrogates were analyzed for the initial calibration. The other target Aroclors were analyzed at one concentration level. The instrument GC23 instrument with dual columns DB-XLB and DB-35MS were used in both pesticides and PCB analysis. Samples for PCBs were analyzed separately from the pesticides.

Continuing Calibrations - Acceptable

All of the continuing calibration verification standards (CCVs) associated with the samples met the criteria for frequency of analysis, the applicable recovery criteria, ion abundance ratios, RT windows, chromatographic resolutions, percent differences (%D) and/or relative percent differences (RPDs) between the initial calibrations' mean and daily instrument response or calibration factors. None of the data were qualified on this basis.

Compound Identification

All of the target compounds detected for each suite of compounds met the method-specified technical acceptance criteria and were judged to be acceptable with the exception of the following:

SVOCs SIM: The following compounds were qualified as tentatively identified at estimated concentrations, "JN", due to weak or incomplete spectra.

- dibenzofuran in samples LWG0103R014TSLSWBC10LWG0105R006TSLSWBC00 and LWG0107R009TSLSWBC00
- bis-(2-chloroethyl) ether in sample LWG0109R006TSLSWBC00

Pesticides: The following single-component pesticides co-elute in the DB-XLB and/or DB-35MS columns. Note: The RT windows used for both columns were calculated by the reviewer by averaging the RTs from the 4/09/03, 4/16/03 and 4/19/03 continuing calibration runs ± 0.07 minutes:

Compounds	DB-XLB RT *	DB-35MS RT *	Action
B-BHC	9.71 - 9.85	9.89 - 9.93	Report detected values off DB-35MS
heptachlor	9.77 - 9.91	7.89 - 8.03	OK, all non-detects
oxychlordane	10.96 -11.07	9.03 - 9.17	Report detected values off DB-35MS
heptachlor epoxide	10.98- 11.12	9.19- 9.33	Report detected values off DB-35MS
2,4'-DDD	11.96- 12.09	10.35-10.49	Report detected values off DB-35MS
dieldrin	11.99- 12.13	10.16-10.32	Report detected values off DB-35MS
g-chlordane	11.47-11.61	9.55 -9.69	OK detected values reported off DB-XLB
trans-nonachlor	11.62- 11.76	9.59 - 9.73	Report detected values off DB-35MS
a-chlordane	11.55 -11.69	9.72 - 9.84	Report with endosulfan I
endosulfan I	11.59- 11.73	9.75 - 9.89	Report with a-chlordane

SVOCs SIM: This analysis was designed to get lower reporting limits for PAH's and other SVOC compounds. All of the samples for this analysis went through special extract clean-up techniques prior to analysis. The laboratory had problems with phthalate contamination in both SVOC full scan and SVOC SIM analyses. This phthalate contamination problem will be discussed further in the Blanks section of this validation report. The laboratory analyzed the samples in accordance with the method/SOP specified in the QAPP.

Butyl Tins GC/FPD: The reporting limits and ACGs listed for butyl tins in the QAPP were met by all analyses.

% lipids: This parameter was performed in accordance with the PSEP protocols. % lipids of the samples ranged from 2-6%.

Instrument Performance

All of the instruments used in the analyses met the instrument performance criteria, the calibration requirements, the established retention time (RT) windows and the analytical sequence specified by the analytical methods. All of the samples were analyzed during an acceptable 10-12 hour analytical period and the instruments used remained stable throughout the course of analyses as indicated by the QC standards analyzed at the end of each period. None of the data were qualified on this basis.

Initial Calibrations - Acceptable

The initial calibrations performed for each suite of parameters met the technical acceptance criteria and the required frequency of analysis specified in the analytical methods. The initial calibrations included the analysis of at least one low standard at the laboratory's Method Reporting Limits (MRL). None of the data were qualified on this basis.

Metals using ICP-AES: Chromium and aluminum (for sample LWG01FZ0609TSBBWBC10) were analyzed using the ICP-AES Method 6010B. The ICP-AES instrument was initially calibrated according to method specifications. The initial calibration verification (ICVs) standards analyzed had acceptable recoveries ranging from 90-110%. No discrepancies were noted in the raw data submitted.

Metals using GFAA: Selenium was analyzed using SW846 Method 7740 and mercury used Method 7471. The instruments used were initially calibrated with a blank and standards at 4 concentration levels. The initial calibration curves for both analytes were acceptable with correlation coefficients >0.995. The ICVs analyzed had acceptable recoveries ranging from 93-107%.

Metals Using ICP/MS: The rest of metals target list were analyzed using Method 6020. The instrument used was initially calibrated. ICVs for ICP/MS ranged from 90-110%.

Organics Analysis (SVOCs, SIM SVOCs, Pests, Butyl tins): The frequency of analysis, the concentration levels, the percent relative standard deviations (%RSDs), the minimum response factors, percent endrin and DDT breakdowns and RT criteria set by the methods were met by all of the initial calibration curves analyzed for the above-listed suite of parameters.

Compounds	DB-XLB RT *	DB-35MS RT *	Action
cis - nonachlor	12.58-12.77	10.75-10.87	Also co-elutes with Aroclors Action will be discussed in the next paragraph
2,4'-DDT	12.41- 12.55	10.75-10.87	OK, detected values off DB-XLB
4,4'-DDD	12.59- 12.73	10.92-11.06	Also co-elutes with Aroclors. Action will be discussed in the next paragraph

The following single-component pesticides co-elute with the Aroclors 1248, 1254 and/or 1260 peaks in both columns:

- cis-nonachlor, 4,4'-DDD, 4,4'-DDT, endosulfan sulfate, methoxychlor, endrin ketone and mirex.

Action: Use professional judgment in qualifying detected cis-nonachlor, 4,4'-DDD, 4,4'-DDT, endosulfan sulfate, mirex methoxychlor, and endrin ketone. If Aroclors are present in the samples, comparable concentrations of detected cis-nonachlor, 4,4'-DDD, 4,4'-DDT, endosulfan sulfate, mirex methoxychlor, and endrin ketone may be attributable to the PCB peaks. In instances like this, the compounds are qualified as non-detects, "UJ" with estimated reporting limits raised at the level of detection due to PCB interferences. Detected compounds listed above at significantly higher concentrations than the Aroclors detected in the samples were qualified as estimated, "J". Corrections on the reporting limits and concentrations of these single-component pesticides in the affected samples were made by the reviewer on their Forms I.

PCBs as Aroclors: The PCBs reported for the most of the samples were weathered and complete isolation of peaks for specific PCB calculations could not be performed. In cases like this, the reviewer re-calculated the results and reported the concentrations as an average of PCB 1248 and 1260 or PCBs 1248/1254/1260. Samples with predominantly high levels of PCB 1260 compared to other Aroclors were reported as PCB 1260 by the reviewer. Corrections on the detected concentrations of Aroclors in the samples were made the reviewer on the Forms I.

Blanks

The frequency of analysis of laboratory blanks was met for all suite of parameters with the exception of the following:

- Metals: Aluminum, antimony, lead, nickel, silver, and thallium were detected in the preparation, initial and/or continuing calibration blanks at concentrations less than the reporting limits. Detected aluminum, antimony, lead, nickel, silver, and thallium in the associated samples at concentrations that were less than 5 times the value in their respective blank(s) were qualified as non-detects, "U".
- SVOC full scan 8270 and SVOC SIM: The SVOC SIM and SVOC full scan used the same method blanks. Acenaphthene and benzo(g,h,i)perylene were detected in the SIM SVOC method blank at concentration levels that were less than the reporting limits.

Detected acenaphthene and benzo(g,h,i)perylene in the associated samples at concentrations less than five times the value in the blank were qualified as non-detects, "U". Bis(2-ethylhexyl)phthalate was detected in both SVOC SIM and SVOC full scan method blanks at concentration greater than the reporting limits. Bis(2-ethylhexyl)phthalate detected in the associated samples at concentrations less than ten times the value in their associated blank were qualified as non-detects "U" with the quantitation limits raised at the level of detection. It is recommended that data users utilize the bis(2-ethylhexyl)phthalate reported off the SVOC full scan results.

Internal Standards (IS) Areas and Retention Times

All of the IS areas and retention times in the SVOCs full scan and SVOC SIM analyses met the method-specified area and retention time criteria. None of the data were qualified on this basis.

Laboratory Control Samples/Duplicate (LCS/LCSD)

The frequency of analysis and the project-required recovery criteria were met by all of the LCS and LCSD sets analyzed for each suite of parameters with the exception of the following:

- Pesticides: KWG0304878-3 and KWG0304878-4 - the RPD for endrin aldehyde (70%) exceeded the control limits (40%).

None of the reported data results were qualified on this basis.

Surrogate Recoveries

SVOC and 8270C PAH SIM Analysis: The surrogate compounds (CLP list) specified in the QAPP were used as surrogates for the SVOC full scan and SVOC SIM. D-10-Methylnaphthalene and d-14-dibenzo(a,h) anthracene was not used as surrogates for SVOC SIM analyses as specified in the QAPP (see Table A7-7). All of the surrogate recoveries were met except for the 2-fluorophenol and 2,4,6-tribromophenol recoveries in the Method Blank which were significantly low. Since all of the surrogate recoveries for both SVOC full scan and SVOC SIM analyses were acceptable for all the samples, none of the SVOC full scan or SVOC SIM data were qualified on this basis.

Pest/PCB Analyses: The pest and PCB analyses used the same surrogates: tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB). The laboratory also used isodrin as injection standard. All of the samples and QC samples met the pests and PCB project-required surrogate recovery limits (see Table A7-7 of the QAPP). None of the PCB and pesticide data were qualified on this basis.

Butyl Tins: Tri-n-propyl tin was used as a surrogate for this analysis. All of the recoveries were within the QAPP specified control limits and were acceptable. None of the butyl tin data were qualified on this basis.

Standard Reference Material (SRM)

The frequency of analysis and the recovery limits were met by all of the SRMs analyzed for each suite of

parameters. None of the reported data results were qualified on this basis.

Matrix Spike and Matrix Spike Duplicate (MS/MSD)

The following samples were designated for QC (MS/MSD) analyses:

LWG0103R002TSSPWBC10 for SVOCs full scan and SVOC SIM, LWG01FZ0609TSBBWBC10 for metals including mercury, pests and PCBs, and LWG0106R002TSCAWBC00 for butyl tins.

SVOC Full Scan Analysis: The routine CLP spike compounds with the addition of benzo(g,h,i) perylene and di-n-octylphthalate were used for MS/MSD analysis for this project. All of the spike recoveries and RPDs were within the project-required control limits (see Table A7-7 of the QAPP). None of the SVOC data were qualified on this basis.

SVOC SIM Analysis: Based on the QAPP, phenanthrene, chrysene and benzo(k)fluoranthene should be used as spike compounds for MS/MSD SVOC SIM analyses. The laboratory used the same spike compounds used in SVOC full scan for MS and MSD analyses. Recoveries were reported for acenaphthene, pyrene, 1,4-dichlorobenzene, 1,2,4- trichlorobenzene, pentachloropehnol, 2,4-dinitrotoluene, N-nitroso-di-n-propylamine and 2-chlorophenol which were all within the QAPP specified control limits (see Table A7-7 of the QAPP). Phenol and 4-nitrophenol were lost during the silica gel clean-up process. Because phenol and 4-nitrophenol results were calculated off the SVOC full scan analysis, none of the phenol and 4-nitrophenol results in the associated samples were qualified on this basis.

Pest Analysis: The pesticide spike compounds listed in the QAPP (table A7-7) plus toxaphene and hexachlorobenzene were used by the lab as spike compounds for MS/MSD analysis. All of the MS/MSD spike recoveries and RPDs were within the project-required control limits (see Table A7-7 of the QAPP) except for the 4,4'-DDE and 4,4'-DDT. The 4,4'-DDT and 4,4'-DDE recoveries could not be accurately determined due to high levels of 4,4'-DDT and 4,4'-DDE native to the sample. None of the data were qualified on this basis.

PCB Analysis: Aroclors 1016 and 1260 were used as the spike compound for PCB analyses. All of the PCB MS/MSD recoveries and RPDs were within the project-required control limits (see Table A7-7 of the QAPP) with the exception of the PCB recoveries for Batch QCMS KWG0304879-1, KWG0304879-2, KWG03066518-1 and KWGG03066518-2 due to the interferences caused by the mixtures of PCBs native to the QC sample. None of the associated PCB data were qualified on this basis.

Butyl Tins: All of the spike recoveries and RPDs were within the QAPP specified control limits and were acceptable. None of the data were qualified on this basis.

Metals Analysis: The frequency of analysis and the recovery criteria were met by all of the metals analyses including mercury. None of the metals data were qualified on this basis.

Duplicate/Triplicate Sample Analysis

Metals: All of the initial and duplicate analytical results were within the project-required RPD limits.

None of the data were qualified on this basis.

Percent Lipids: The duplicate analysis met the QAPP specified technical acceptance criteria for this parameter. None of the data were qualified on this basis.

Analytical Sequence - Acceptable

All of the standards, blanks, samples and QC samples for each suite of parameters were analyzed in accordance with the method- specified analytical sequence. None of the data were qualified on this basis.

Serial Dilution

All of the compounds detected at concentrations that were >50x the MRL met the project-required %D limits with the exception of aluminum (19%) and nickel (18%). The aluminum and nickel reported in all of the samples were qualified as estimated, "J".

ICP Interference Check Samples (ICS) - Acceptable

The frequency of analysis and recovery limits for ICSAB were met. The ICSAB recoveries ranged from 89 - 110%. None of the data were qualified on this basis.

Laboratory Contact

Striplin Environmental and the laboratories were not contacted for this data validation.

Overall Assessment

With the exception of the PCB and pesticides data, the SVOC full scan, SVOCs SIM, metals, butyl tins and % lipids may be partially validated using the summary of QC forms. Pesticides and PCB data should be fully validated and validators should check the validity of the detection of PCB Aroclors (or mixtures of PCB Aroclors), DDT and its metabolites and some single-component pesticides. Co-elution of single-component pesticides also needs to be evaluated. It is highly recommended that a reverse search of SVOC TICs be also conducted by the SVOC lab for samples with high concentration (ppm levels) detections of DDT and its metabolites and PCBs.

There were no significant problems encountered in this validation. All of the samples were analyzed in accordance with the method, SOP and QAPP specifications. The data, as qualified, can be used for all purposes.

Data Qualifiers		
	U	The analyte was not detected at or above the reported numeric result.
	J	The analyte was positively identified. The associated numerical result is an estimate.
	UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
	R	The data are unusable for all purposes.
	N	There is evidence the analyte is present in this sample.
	JN	There is evidence that the analyte is present. The associated numerical result is an estimate.